

BEST AVAILABLE COPY

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2002-192652

(43)Date of publication of application : 10.07.2002

(51)Int.Cl.

B32B 15/08

C08J 5/18

C08K 3/04

C08K 7/04

C08L 79/08

C23C 28/02

C25D 5/56

C25D 7/00

H05K 1/03

(21)Application number : 2000-395454

(71)Applicant : GUNZE LTD

(22)Date of filing : 26.12.2000

(72)Inventor : NISHIURA NAOKI  
NODA KAZUHIRO

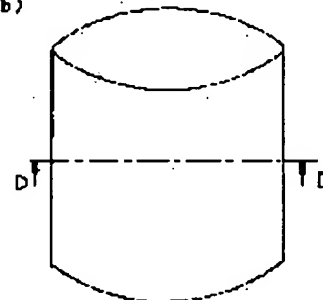
## (54) METALLIZED POLYIMIDE FILM

## (57)Abstract:

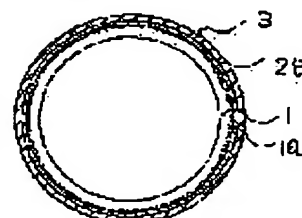
**PROBLEM TO BE SOLVED:** To provide a polyimide web-like or endless pipe-like film, with a conductive metallized surface, which is highly adhesive even at high temperature.

**SOLUTION:** A polyimide film (1) has a powder A of a fine fiber-like inorganic compound, with a coefficient of linear expansion equal to or below that of a polyimide resin, which is unevenly distributed and dispersed mostly in the surface part. In addition, the metallized polyimide film is constituted of a conductive thin metal film layer (2B) formed of a conductive metal B by a physical thin film forming means and a conductive thick metal film layer (3) formed by an electroplating process, coherently laminated, in that order, on the surface of the polyimide film. When the film is in the web shape, it can be used, for example, as an FPC substrate and when the film is in the shape of an endless pipe, it can be used, for example, as a heating belt for the electromagnetic induction process of a copying machine.

( : b )



(1 a)



D : 断面

## LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of

**\* NOTICES \***

**JPO and NCIPi are not responsible for any damages caused by the use of this translation.**

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**CLAIMS**

---

[Claim(s)]

[Claim 1] It is the polyimide system film (1) with which it comes to carry out many maldistribution distributions into a surface part of the microfilament-like inorganic compound fine particles A which have the coefficient of linear expansion below the coefficient of linear expansion which polyimide system resin has. And the metalization polyimide system film which the sequential adhesion laminating of this film front face is carried out, it consists of a conductive metal thin film layer (2B) formed of the physical thin film means forming of the conductive metal B, and a conductive metal thickness membrane layer (3) formed by electrolysis plating, and is characterized by things.

[Claim 2] The metalization polyimide system film according to claim 1 said whose microfilament-like inorganic compound fine particles A are the inorganic compound fine particles of the shape of a whisker of specific gravity 2.5-4.

[Claim 3] The metalization polyimide system film according to claim 1 or 2 with which said microfilament-like inorganic compound fine particles A were chosen from the group of whisker-like potassium titanate, boric-acid aluminum, silicon carbide, silicon nitride, and an alumina and which is a kind at least.

[Claim 4] A metalization polyimide system film given in any 1 term of claims 1-3 which said microfilament-like inorganic compound fine particles A come to contain two to 10% of the weight to polyimide system resin.

[Claim 5] A metalization polyimide system film given in any 1 term of claims 1-4 said whose polyimide system films (1) are non-edge pipe-like polyimide system films.

[Claim 6] The metalization polyimide system film according to claim 5 characterized by coming to also contain conductive carbon black fine particles on said non-edge pipe-like polyimide system film in the state of homogeneity distribution substantially.

[Claim 7] The metalization polyimide system film according to claim 6 which said conductive carbon black fine particles come to contain five to 15% of the weight to polyimide system resin.

[Claim 8] A metalization polyimide system film given in any 1 term of claims 1-7 which copper, nickel or aluminum, and a conductive metal thickness membrane layer (3) become [ said conductive metal B ] from any one sort of copper or nickel.

[Claim 9] A metalization polyimide system film given in any 1 term of claims 1-8 which said physical thin film means forming becomes by the sputtering method which uses the conductive metal B under 80-160 degrees C as a target.

[Claim 10] A metalization polyimide system film given in any 1 term of claims 1-9 which the thickness of 100-1000nm and a conductive metal thickness membrane layer (3) becomes [ the thickness of said conductive metal thin film layer (2B) ] from 1-50 micrometers.

---

[Translation done.]

**\* NOTICES \***

**JPO and NCIPi are not responsible for any damages caused by the use of this translation.**

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**DETAILED DESCRIPTION**

---

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the improved metalization polyimide system film. This film is used effective in for example, the substrate for FPC (flexible print), the member for copying machines, etc.

[0002]

[Description of the Prior Art] Since it has the property in which polyimide was variously excellent as compared with other polymers, it is going across the application in many fields. Especially, adhesion compound of copper foil and the polyimide film is carried out, and there is much use as a FPC (flexible print) substrate. Moreover, recently, the use as a member of the belt-like photo conductor of the color copying machine which took in the middle imprint method, and a belt-like middle imprint object is also considered, and practical use has also been carried out in part.

[0003] It is important in a FPC substrate to say how thinner copper foil is powerfully pasted up on a polyimide film. Although it is satisfactory in respect of this adhesion, it is a fault that there is whenever [ industrial-engineering upper limit ] about the thickness in the case of this copper foil. then -- or it vapor-deposits copper with vacuum deposition in order to solve the point of this thickness, or it plates copper with plating (radio plating-electrolytic plating) -- being certain -- it is -- both this \*\* is combined, or a thing means is examined, and much patent application is also made. Although this means is effective at the point that moreover thickness is freely controllable, more thinly than copper foil, the problem is the point of the adhesion force with this film. Examination is wholeheartedly made also about the means for this adhesion force improvement, and much patent application is made and proposed also about this. However, the actual condition is that the FPC (ultra-thin copper-clad) substrate which comes to have adhesion force sufficient in the present condition although it is usable also to the use in an elevated temperature also to repetitive folding, for example is not obtained.

[0004]

[Problem(s) to be Solved by the Invention] Then, adhesion formation was carried out by the ultra-thin thickness for which conductive metals, such as copper, ask freely to a polyimide film, and this invention person etc. has inquired wholeheartedly by making into a technical problem to develop the metalization polyimide film improved by even practical use level, without exfoliating, even if used by the shape of a belt which performs repetitive bending actuation further even if this is used at an elevated temperature. As a result, the solution means could be found out at last, and this invention was reached. Invention found out in this way is indicated for the following solution means.

[0005]

[Means for Solving the Problem] Namely, this invention is a metalization polyimide system film (it is called a M-PI film below) indicated to claim 1. It is a polyimide system film (1) with which it comes to carry out many maldistribution distributions into a surface part of the microfilament-like inorganic compound fine particles A (for it to only be called the microfilament fine particles A below) which have the coefficient of linear expansion below the coefficient of linear expansion which polyimide system resin (it is called PI resin below) has. And the sequential adhesion laminating of this film front face is carried out, it consists of a conductive metal thin film layer (2B) formed of the physical thin film means forming of the conductive metal B, and a conductive metal thickness membrane layer (3) formed by electrolysis plating, and is characterized by things.

[0006] And invention indicated to claims 2-10 in relation to said claim 1 is offered, and above all, the gestalt of the metalization polyimide system film of claim 1 is invention to a non-edge pipe-like film, and what

semi-conductivity also gave this especially whole film base by conductive carbon black doubles and offers claims 6 and 7. Thus, semi-conductivity can also be given to the metalization endless tubular polyimide system film which is one of the claims 1 by having semi-conductivity. It comes to be able to perform electrification from a rear face by having this semi-conductivity. The use as a middle imprint belt member of a color copying machine is also attained, and, as for this, is also that versatility is expanded further. Although this invention is as aforementioned, the following operation gestalt explains it to a detail.

[0007]

[Embodiment of the Invention] PI resin which constitutes the base of the M-PI film which is this invention first says the aromatic series PI resin and aromatic series polyamidoimide (PAI resin) which have the thermosetting or thermoplasticity generally known, is suitably chosen from these and is used. Therefore, there is especially no limit in the aromatic series tetracarboxylic dianhydride which is the start raw material of PI resin, aromatic series tricarboxylic acid 1 anhydride in the case of aromatic series diamine and PAI resin, the class of aromatic series diamine and the combination of each raw material of this, and its number. You may be blend PI resin with which these resin was blended suitably, of course.

[0008] As for thermosetting or thermoplasticity, said PI resin is decided by the class of start raw material. That is, if this raw material with which two or more -O-, -SO<sub>2</sub>-, -CO<sub>2</sub>-, etc. are combined in a polymer principal chain is used, it will be easy to become thermoplasticity, and it will become thermosetting if these combine one piece or this raw material that it does not have. Generally it is thermoplasticity in on the other hand having already had the amide group about PAI resin. In addition, as special resin, fluorine joint PI resin (for example, thing which has the perfluoroalkyl radical permuted by the polymer principal chain by the fluorine atom) is mentioned. This thing excels other resin in the point of a mold-release characteristic. Although based on the demand characteristics in the application, thermosetting PI resin of what is chosen in each above resin is synthetically good.

[0009] And the gestalt of said PI system resin is a film (long picture) or an endless (with no joint) tubular film offered by claim 5. And distributed content of the microfilament fine particles A which have the low coefficient of linear expansion below the coefficient of linear expansion (coefficient of thermal expansion) which this especially resin has in any case is carried out, and it is said that it comes to be unevenly distributed into the part from which the distributed content condition moreover serves as a front face of this film mostly.

[0010] Although the thickness of PI film (it is called an A-PI film below) with which maldistribution distribution of said microfilament fine particles A is carried out on a front face, width of face, and a diameter are proper by mechanical physical properties (reinforcement, flexibility, etc.), a use gestalt, etc. which this resin itself has, generally they are 600mm or less in the thickness of 50-150 micrometers, width of face of 1000mm or less, and diameter (if it is in a non-edge pipe-like film). In addition, generally about a long film, it is dealt with by the shape of Webb.

[0011] Being specified with the above, especially an A-PI film is based on the following reasons. It explains from the reason the microfilament fine particles A are chosen especially first. although the mechanism of action is not certain, the mixed compatibility of appearing more notably than what adhesion with the conductive metal thin film layer (2B) by the physical thin film means forming mentioned later depends on other means, and PI system resin is comparatively good -- etc. -- it depends. However, also in such a reason, it is further restricted by coefficient of linear expansion. This is because what the adhesion force of a between [ the electric conduction metal layers which become PI system film plane as a base from the conductive metal thin film layer (2B) by which adhesion formation is carried out, and a conductive metal thickness membrane layer (3) ] declines especially on the occasion of use in an elevated-temperature field (for example, 150-200 degrees C), and causes partial exfoliation is lost. It is thought that this effectiveness is because the linear expansion of this film became smaller. Therefore, by these fine particles A beyond the coefficient of linear expansion which PI system resin to be used has, even if it corresponds to the microfilament fine particles A, since the adhesion force becomes a fall inclination, it removes from this invention. Originally, a metal and resin are heterogeneous and coefficient of linear expansion also differs from figures double [ 1-]. furthermore -- an elevated temperature -- increasingly -- the difference of this expansion coefficient of both -- large -- becoming -- just -- being alike -- it becomes a fall operation of the layer adhesion force, and appears. Although similarly considered such a general judgment also in this invention, especially in this invention, such a phenomenon does not happen only within the microfilament fine particles A with the coefficient of linear expansion below PI system resin. This is not certain in what kind of the mechanism of action it is, either.

[0012] And even if it is said microfilament fine particles A specified, it is further regulated by the

distributed condition in the inside of PI film, and it needs to be mostly unevenly distributed into the part used as especially a front face, and it is necessary to distribute. It is saying that it must not never be said that it distributes to homogeneity over the whole thickness direction, or it is mostly [ conversely ] unevenly distributed in a part for a flesh-side surface part, and distributes. This reason is as follows. The adhesion force of the conductive metal thin film layer (2B) by the physical thin film means forming formed in this film plane becomes firmer because it is mostly unevenly distributed into a surface part first. This is considered to be for the adhesion operation between B grains of activity conductivity metals and the microfilament fine particles A which are that the detailed concave convex which already acts effective in adhesion (existence of many microfilament fine particles A) is formed, and the physical thin film means forming of the conductive metal B is taken further there, and jump out by that cause at high speed with the larger front face of this film to work. Moreover, in the microfilament fine particles A existing in a surface part, there is no individual change in the flexibility which the PI system film itself has, and the bad influence to smooth rotation and endurance required as a belt can be prevented. Furthermore, since it is adiabatic toward the front face of this film to the rear face, the heating effectiveness in a front face also becomes good. In addition, although it is that the condition of having distributed in the shape of a layer only on the front face is [ semantics that it is mostly unevenly distributed into the part which serves as a front face here and distributes ] the most desirable, as long as it seems that a bad influence is not greatly done to the operation effectiveness stated to the aforementioned reason, it is saying that some inner dispersion may occur.

[0013] The microfilament fine particles A are specifically as follows. First, these fine particles A are the same as the coefficient of linear expansion which PI system resin has at least especially in coefficient of linear expansion, or need to be smaller than it so that it may describe above. Since the coefficient of linear expansion which PI system resin which serves as criteria on a numeric value has is in the range of  $(9 \times 10^{-5})$  cm/cm/\*\* generally (it changes according to the molecular structure)  $(1 \times 10^{-5})$ , it will call it less than [ this ], but it is more small, and since the effectiveness said above becomes large, it is so desirable that both difference becomes large. Especially the configuration of these fine particles is a microfilament-like, and it is conditions that it must not also be by no means granular. This is because it is not more greatly [ than that the detailed irregularity which appears in a front face is easy to be formed / to be in the condition of being easy to secede from a front face, and / in irregular, and the combination effectiveness with said physical thin film means forming ] discovered in the condition that this carries out many maldistribution distributions on a front face to it being a mere particle. This reason is [0014] which can consider that the condition of a distributed array [ in a front face ], the debt by PI resin, and the gestalt of fine-particles A own [ this ] act conjointly in multiplication (when it is the whisker mentioned especially later) although it is not clear. And the semantics of the fiber of the microfilament fine particles A says the thing of single crystal (mustached crystal) fiber with the diameter of about several micrometers generally called chopped fiber, the thing which is a mere single fiber with a die length of about 1-3mm, a diameter [ of 3nm - several micrometers ] which is made to carry out crystal growth to 1 shaft orientations currently further called the whisker, and is obtained, and a die length of several micrometers - about 500 micrometers. The latter whisker-like fiber is more desirable. As a concrete compound, each whisker, such as the chopped glass fiber which has coefficient of linear expansion in the range of  $10^{-6}$ - $10^{-7}$ cm/cm/degree-C order, for example, potassium titanate, boric-acid aluminum, silicon carbide, silicon nitride, an alumina, and a graphite, is shown. this -- specific gravity -- between 2.5-4 -- it is -- and \*\*\*\*\* -- a high thing is also a desirable reason. As for this, mixed dispersibility with a shaping (it mentions later) raw material is comparatively good, and it is because maldistribution distribution to the surface part discovered with shaping is performed more easily. Furthermore, since effectiveness is promoted more, each whisker of potassium titanate or boric-acid aluminum is desirable. [0015] Although a complement will be told to discovering preferably the operation effectiveness aforementioned by carrying out maldistribution distribution into the surface part of PI film base, if the amount of mixing of the microfilament fine particles A illustrates this concretely, it will be 2 - 10 % of the weight preferably one to 15% of the weight to PI system resin. The adhesion effectiveness by said operation stops discovering this substantially at less than 1 % of the weight. On the other hand, if it exceeds 15 % of the weight, about [ becoming a fall inclination ] and flexibility will fall and this adhesion effectiveness will also be especially depended on saying that a crack becomes easy to enter with a folding use gestalt. Although these fine particles A have as much as possible good little addition, the thing of the shape of said whisker is more effective at this point. Especially in the case of an endless tubing-like film, it is because it is also easy to stand in the shape of a field at a circumferencial direction in a line with this tending to become a detailed concave convex with this film front face advantageous to a bigger adhesion force manifestation.

[0016] Said A-PI film is used by the shape of a non-edge pipe-like film, and in this case, homogeneity distribution can be carried out at the whole and semi-conductivity can also be further given to the non-surface part of this film for conductive carbon black fine particles (following CB fine particles), for example (claims 6 and 7). This is because this film is effective when it is used as the belt-like photo conductor of a color copying machine, and a heating [ a belt-like imprint-cum-] constant member. That is, it is because it becomes easy to carry out the conditional control referred to as \*\*\*\*(ing) timely [ it can be easily charged in the amount needed as it is also at low applied voltage by having semi-conductivity, and ]. Therefore, if the distributed condition that CB fine particles also increase in a surface part is taken, it will come to act in the direction in which the adhesion of about [ that it is hard coming to carry out this operation ] and a conductive metal thin film layer (2B) also worsens.

[0017] Moreover, it is because the operation referred to as that these microfilament fine particles A distribute CB fine particles to homogeneity at the whole at a surface part works more effectively when that said CB fine particles are chosen fabricates in the shape of a non-edge pipe-like film temporarily first with the casting centrifugal method which mixes with the microfilament fine particles A at a shaping raw material, and is mentioned later and it manufactures. Especially bulk density depends this on a very small thing rather than other conductive agents. Moreover, it is because the electric resistance value for which is easy to carry out mixed distribution with a shaping raw material, and it asks in the comparatively little amount of mixing can be acquired. If a volume-resistivity value shows the electric resistance value for which it asks here, it will be 107 - 1010 ohm-cm desirably 102 to 1012 ohm-cm.

[0018] Said CB fine particles have a thing with various physical properties (electric resistance, volatile matter, specific surface area, particle size, a pH value, DBP oil absorption, etc.) according to manufacture raw materials (natural gas, acetylene gas, coal tar, etc.) and manufacture conditions (combustion conditions). It is good the high thing of CB fine particles from which a lower electric resistance value is easily acquired as the mixed, little possible distribution is also, for example, the electric conduction index with which structure progressed, and to choose CB fine particles which contain many volatile components which make a pH value low, although (CB fine particles which manufactured acetylene gas as a raw material and obtained it having much this), or an electric conduction index is not so high. However, since it may be easy to generate variation in the electric resistance value given, CB fine particles from which a lower electric resistance value is acquired by little addition are good to choose proper CB fine particles so that it may check beforehand [ the / point ] enough and trouble may not be caused to it. If the concrete amount of mixing is illustrated from this semantics, it will be 5 - 15 % of the weight preferably three to 20% of the weight to PI system resin.

[0019] Although it is as [ film / said / A-PI ] aforementioned, the manufacture means of this film is explained here.

[0020] PI system resin solution which is a manufacture raw material (shaping raw material) first is prepared. It differs in the case where PI system resin is thermoplastic PI resin, PAI resin, and thermosetting PI resin here. Generally, a front, since two sorts have the property dissolved in an organic solvent even if they are imide-ized, they carry out the polymerization reaction of this molar quantity of at least 2 components (aromatic series tetracarboxylic dianhydride or aromatic series tricarboxylic acid 1 anhydride, and aromatic series diamine) in an organic solvent, advance it to imide-izing, and are obtained as thermoplastic PI resin solution or a PAI resin solution at once. However, since it will become insoluble to an organic solvent if a reaction is advanced to imide-izing in the case of the latter, it is not obtained as a thermosetting PI resin solution at once. In this case, it is necessary to stop a reaction and to obtain as a PA acid solution in the phase of that precursor (following PA acid), i.e., a polyamide acid.

[0021] Although it is necessary to advance a reaction until the physical properties which were excellent as a film result in sufficient polymerization degree to be obtained, even if it makes it thermoplastic PI resin, PAI resin, or PA acid, it will be said that each solution concentration (that is, solid content concentration) obtained is obtained by the convenient concentration for shaping. What is necessary is to dilute with the same solvent as the organic solvent used at the reaction, and just to adjust, when the concentration temporarily obtained at the reaction is high. Generally solution viscosity is good to adjust in the range of 0.5-5Pa and s. In addition, said organic solvent is an organic polar solvent of non-proton systems, such as N-methyl pyrrolidone (henceforth, NMP) and dimethylacetamide which are generally known as a solvent of PI system resin, and dimethyl sulfo oxide.

[0022] Next, although the microfilament fine particles A, or these fine particles A and CB fine particles of the specified quantity are added by said obtained PI system resin solution (following virgin undiluted solution) and mixed distribution is carried out, in order to improve dispersibility at this time, the surfactant



of a fluorine system etc. may be added, but even if it adds, it is good to stop about 1 or less % of the weight to this resin as a little as possible. A mixed procedure is good to perform it as follows. If it adds first, it will mix enough preparatorily by the mixer with an impeller. And this reserve mixed liquor is moved and changed to a ball (product made from ceramics) mill stirring mixer, and mixed distribution is carried out further. Distributed mixing is fully carried out by this two-step mixing. When you need degassing, it is good to carry out promptly, stirring slowly.

[0023] And although said prepared manufacture undiluted solution is fabricated by a long film or the non-edge pipe-like film, it is that microfilament fine particles carry out many maldistribution distributions into a surface part of the main process condition here, and the homogeneity distribution of the CB fine particles is made to be carried out at the whole. To it, the following approaches can illustrate preferably.

[0024] It explains from a long film first. although first put into said manufacture undiluted solution and virgin undiluted solution by two tanks with an impeller -- as a manufacturing installation -- an outline -- the following can be illustrated. Each tank is connected with two slit-like nozzles through a flexible tube, and is arranged on a metal belt. this -- two nozzles keep predetermined distance to this belt, and although it is parallel and is installed horizontally, this nozzle for manufacture undiluted solutions is back arranged to the travelling direction of this belt. Although the heating means is attached to this metal belt mainly for organic solvent evaporation, whenever [ stoving temperature ] is controlled to become high gradual gradually. The die length of this metal belt is proper mainly by relation with a shaping rate. In addition, it is good to be always made to stir by the tank for manufacture undiluted solutions especially. This is for always maintaining the microfilament fine particles A at a homogeneity distribution condition.

[0025] And the discharge paint film of the manufacture undiluted solution is carried out to said metal belt side which carries out rotation advance with constant speed by predetermined discharge quantity from a slit nozzle. Although the regurgitation in this case may be liquefied, the letter of spraying is sufficient. The latter is good for control of thinner thickness. Next, the discharge laminating of the virgin undiluted solution is carried out to said paint film side by predetermined discharge quantity from this nozzle for virgin undiluted solutions. Although the timing of the latter regurgitation is shifted, as for the time amount to shift, it is good that it is the time amount from which the front face of the manufacture undiluted solution by which the paint film was carried out will be got dry a little. although discharge quantity of a manufacture undiluted solution is lessened here as compared with it of a virgin undiluted solution, it is set up so that it may become less than 15% of the overall thickness of for example, a last profit \*\*\*\* A-PI film. It is from the reason said that this is mostly unevenly distributed into the surface part described above. Although the organic solvent which the paint film on this belt by which the laminating was carried out is gradually heated by the elevated temperature, and is contained advances by carrying out evaporation \*\*\*\* gradually, the \*\*\*\*\* is good as about 70 - 90% to make it remain 10 to 30%. This is because it is connected with the dry area of a film plane etc., so it is not desirable to remove these all solvents at once on this belt. Therefore, temperature here is good to carry out stairway-change within the limits of about 100-200 degrees C. Moreover, although it is necessary to perform imide-ization other than evaporation \*\*\*\* of this solvent, since this imide-ization is performed more at an elevated temperature (for example, 250-400 degrees C), on this belt, it is good in PA acid use, to carry out through the tunnel-like hot-air-drying furnace of separate installation etc., without carrying out.

[0026] or [ and / once rolling round here, the free-standing film on said dried metal belt exfoliating ] -- or it passes through the inside of the tunnel-like hot-air-drying furnace formed separately continuously continuously, evaporation removal of all the residual solvents is carried out, and a desired A-PI long picture film is manufactured. Since it is necessary to carry out to imide-izing about the thing of PA acid use here, temperature control is made to be performed with a temperature gradient so that the temperature requirement in this case may become high as range of 100-450 degrees C as the temperature of this dryer goes to an outlet.

[0027] Next, the shaping approach of an endless tubing-like A-PI film is explained. In order to fabricate in the shape of endless tubing first, the casting centrifugal method generally known is used preferably. that is, the inner skin of the metal drum which rotates with the rotational speed on which a centrifugal force acts at least -- using -- shaping \*\*\*\*\* -- as the equipment -- an outline -- a degree can be illustrated. The metal drum (mirror plane finishing by the chrome plating by which about  $R_z=0.5$ micrometer was made to the inside) of both-ends opening is laid on two rotation rollers (attachment and detachment are free). This drum takes the device which carries out indirect rotation by rotation of this roller. And the source of heating for heating the inside of this drum (for example, far infrared rays) is established in the outside upper part. The source of heating is prepared also in this roller here, and auxiliary heating of this drum is performed. And in

this drum, the slit-like nozzle for shaping undiluted solution regurgitation which isolates from this field between predetermined, and moves horizontally horizontally, and comes to have the device it can be inserted [ device ] is prepared. The outlet width of face (slit width) of this nozzle was called about 0.2-3mm, and it just called die length about 10-100mm. Thereby, supply width of face and the amount of supply (spreading thickness) are decided. And this whole drum at least is promptly removed out of a system in the organic solvent by which is surrounded with a case with a ventilating fan and heating evaporation is carried out during rotational casting. Of course, even if a computer is incorporated so that the film thickness for which it asks may be obtained freely, and the rate of the amount of supply of a shaping undiluted solution, the rotational speed of this drum, the moving rightward of this nozzle, or \*\*\*\* may be controlled automatically, it is.

[0028] and the shaping procedure by said shaping equipment -- an outline -- it is done as follows. First, said nozzle detaches about 30-50mm in the upper location at the right end of [ inner ] a metal drum, and is arranged in it. And this drum begins rotation with a predetermined rotational speed by which computer control was carried out. An important thing moves the microfilament fine particles A so that it may be mostly [ promptly ] unevenly distributed into a surface part, and CB fine particles are controlling rotational speed as homogeneity's distributes at the whole in the original state here. Since a big centrifugal force will be applied at once if rotational speed is made high at this also at remainder, CB fine particles also come to move to a surface part. It is good to carry out with the rotational speed which gets to know this and maintains both balance through preliminary experiment suitably. In preliminary experiment, it is checking that it can attain if it is the range of about 5-30 rads/s. If rotation of a metal drum starts, the migration at a left end from the right end of this nozzle will start under computer control at the same time supply of the shaping undiluted solution from a slit nozzle starts. If the supply at a left end from a right end finishes, injection supply will be suspended immediately, and a original location is made to once carry out the auto return of this nozzle, it is retreated further, and is taken out out of a system. Next, the metal drum which is continuing rotating is surrounded with a case, heating by said source of heating is started, and the inside of this drum is maintained at predetermined temperature. Operation of the ventilating fan of said case is also started with initiation of this heating. Rotational speed at this time may be carried out to the case of being the same as the original rate, quickly further a little or late (generally about 0.5 to 2 times of said first rate). Fundamentally, although heating conditions here are higher than the evaporation temperature of an organic solvent, they are made into temperature (about 100-200 degrees C) lower than imide-ized temperature (about 250-450 degrees C). This has the semantics [ an electric resistance value ] of stability maintenance in the thing using PA acid at some by which CB fine particles also carry out content distribution of the imide-izing although it is because it cannot progress substantially in order to make the one section remain without removing no organic solvents so that it may describe above also here.

[0029] If shaping on said metal drum finishes, when it secedes from this drum to a non-edge pipe-like film be [ it / this drum / continue ], and it puts into the hot air drying equipment of separate installation and it is based on PA acid with removal of the organic solvent which remains, imide-ization will be performed and an endless tubing-like A-PI film will be obtained. Therefore, although hot blast temperature is an about 100-450-degree C temperature requirement, it is good to have faced the temperature up, to carry out the temperature up also of the case of a gap in gradual stairway, and to carry out fixed time amount heating at the temperature. Let hot blast be a heating medium especially here for the generated residual solvent and the condensation water generated in the case of imide-izing removing out of a system promptly compared with mere heating. The adhesion force of the conductive metal thin film layer (2B) mentioned later as a result is also referred to as being also in the surface state which becomes stronger.

[0030] In addition, the difference with the case where break away with the case where hot blast heating is carried out with this drum equipped (\*\* which does not secede from said metal drum to a non-edge pipe-like film), and hot blast heating is carried out is based on PI system resin which is a matrix. That is, it is saying that it once breaks away, this resin (for example, PI resin combined with a principal chain only by the benzene nucleus which does not have ether linkage etc.) that has a contraction inclination along with evaporation of a residual organic solvent is heated, and this resin (for example, PI resin which has ether linkage etc. in a principal chain) that is not so is heated with this drum. About this film that once breaks away here and is heated, it is good not to put into a dryer as it is, to fit this in hollow tubular metal mold (for it to be the outer diameter of eye small \*\* a little from the bore of a film), to put this metal mold into a dryer and to heat it. This is for regulating contraction.

[0031] And for surface metalization, first, as for the A-PI film which fabricated as aforementioned and was obtained, a conductive metal thin film layer (2B) is formed as the conductive metal B is also. that is, the



vacuum deposition method with which this means is generally known although this especially thin film layer is formed of the physical thin film means forming of the conductive metal B and ion play TENGU -- either law or the sputtering method is said. The sputtering method is effective especially above all. that is, the thing for which this means is used -- what kind of other means (for example, an electroless deposition method --) The adhesion force with this film plane is higher than chemical or the surface activity-ized method by the physical method. And engine-performance quality (purity, precision, low electric resistance, etc.) of a layer own [ this ] is good, and formation of the conductive metal thickness membrane layer (3) by the electrolytic plating which is another means further is also performed smoothly, and it is said that the metal thickness film with the high adhesion force is formed easily. In addition, it is assumed here that the effectiveness over the adhesion force with this film plane being good is based on the following mechanisms of action. the result to which the molecule (activity) of the conductive metal B which jumped out at high speed attacks the microfilament fine particles A which exist (protrusion) on this film front face, and carries out light association chemically (physics) -- coming out -- isn't it?

[0032] Although it is the simple substance metal which flows quickly also with the current of \*\* and a minute amount as said conductive metal B, or its alloy, as the electric resistance value, it is as follows [ abbreviation 10-4ohm/\*\* ]. Each alloy which specifically makes a principal component copper, nickel, silver, platinum, tin, KADONIUMU, aluminum, or these can be illustrated. The more desirable thing in respect of the adhesion force is copper, nickel, or aluminum above all. It is also for using as an effective electrode in the case of the electrolytic plating performed to a degree that conductivity is required here.

[0033] It is decided in consideration of carrying out a bad influence to it being a thin layer also in the adhesion force also at minimum thickness required for the thickness of a basing-on said conductive metal B thin film layer acting effectively as said electrode, and remainder, and this thin film stratification plane having said that it was better to have become in the smooth side. If it illustrates from this point, 100-1000nm will be 300-700nm preferably.

[0034] The formation conditions of the all directions method illustrated by said physical thin film means forming are illustrated about the sputtering method mentioned as a desirable approach here, although it can carry out while being used generally. When an A-PI film is a long film first, it rolls round in the state of the Webb volume, and arranges with a roller in the vacuum chamber (spatter room) of spatter (cathode is planar mold magnetron) equipment. This film plane that has the microfilament fine particles A on a front face keeps predetermined distance, and is arranged towards the conductive metal B target on a planar. Generally a pressure is good in an argon permutation at the time of the spatter in a vacuum chamber 3 - 15 W/cm<sup>2</sup> (DC and 0.2-0.5kv) extent and for an electrical-potential-difference current to make [ 80-160-degree C ] especially preferably spatter temperature (maintenance temperature in a vacuum chamber) 90-140 degrees C at the time of 10<sup>-3</sup> to ten to 1 Pa, and a spatter. Since a sputtering rate (rolling-up rate) is decided by thickness of electrical-potential-difference current strength and the thin film layer (2B) to desire at the time of a spatter, it is good about this to decide with a prior check. Even if a M-PI film is used at nearly 200 degrees C, spatter temperature (maintenance temperature in a vacuum chamber) will be made into 80-160 degrees C especially here because there is also no risk of a surface metalization layer exfoliating. That is, it is saying that it is effective in grant of heat-resistant adhesion. In addition, it is more desirable to carry out, in order to acquire the adhesion about which is more trustworthy and he can feel easy, although it is not necessary to perform pretreatment (chemical or physical processing) generally performed by especially an A-PI film being used. This pretreatment and a chemical twist have a desirable physical method by plasma electrodischarge treatment, sandblasting, etc. the microfilament fine particles A of this film plane receive a damage by the plasma, and this leads to improvement in the further adhesion -- coming out -- it is thought whether there is nothing.

[0035] And sputtering in case an A-PI film is a non-edge pipe-like is performed preferably as follows. First, at the time of pretreatment and a spatter, at the time of a pressure, temperature, and a spatter, a configuration is a non-edge pipe-like film, and although carried out in the range illustrated above about the conditions of an electrical-potential-difference current, in order to carry out the spatter of two or more [ of this ] at once, the following devices are taken. First, a vacuum chamber is made into the shape of a drum of a vertical mold, and the wall of this drum arranges one conductive metal B target (cathode) lengthwise. On the other hand in the bottom of this drum, the rotating turntable is prepared, and in pitches [ object / cylinder ], fixed distance is kept [ from / this ] from the rim of this table, and it is confronted each other and set up. This cylinder object is for carrying out fit-in maintenance of this film, and has taken the device which also carries out rotation with rotation of this turntable. If it is made such structure, in order to obtain the thickness of the conductive metal B thin film layer for which it asks, the following two things are made. One of them fixes

rotating velocity of this cylinder object, and it controls this thickness by rotation (a rate and rotational frequency) of this turntable. The two fix rotational speed of this turntable, and they control this thickness by rotation (a rate and rotational frequency) of this cylinder object. In the case of this at least one non-edge pipe-like film, the latter approach is taken although it is decided from relation with the whole processing effectiveness whether to carry out by which approach.

[0036] In addition, when performing glow discharge as pretreatment, this cathode may not be so [ unlike a spatter, distance of a cathode and an A-PI film is made contiguity (for example, 10-20mm), but ], if the same thing as a target may be used. It is more desirable to bring other metals B to a cathode, when a spatter is aluminum. Although a cause is not known well, there is no improvement effect of the adhesion force and it may fall conversely. This glow discharge processing that uses the point, for example, chromium NIUMU, as a cathode is effective.

[0037] Next, on the conductive metal thin film layer (2B) formed in said A-PI film plane, the M-PI film which the laminating of the conductive metal thickness membrane layer (3) is carried out by electrolytic plating, and it makes the purpose by it is formed. Although there is almost nothing that is specified if the conductive metal of a conductive metal thickness membrane layer (3) is a metal which is the good conductor of low resistance and is plated in [ it is efficient and / as this metal thin film layer (2B) ] one rather than the electrical resistivity, the EQC, or it which is said in said conductive metal thin film layer (2B), it is good that they are the same conductive metal as this metal thin film layer (2B) or a good conductor beyond it as one desirable condition. This is because the more excellent printed circuit and electric induction generation of heat can be performed. A desirable thing is the thick-film layer of copper or nickel concretely. In order to plate these metals in this metal thin film layer (2B), the water solution of the inorganic compound of this metal will be used as the electrolytic solution.

[0038] Although it is the need (to for example, the effective manifestation of said printed circuit function and an induction exoergic function sake) to be formed more thickly than that of this thin film layer (2B), since the thickness of said thick-film layer (3) has the point of flexibility, and the danger called exfoliation on the occasion of crookedness use, it is not desirable that it is beyond the need. Since it acts effective [ the thickness of a \*\*\*\* thin film layer ] in electric conduction, the thickness of this thick-film layer can also be set up that much thinly. The appropriate range of 1-50 micrometers is 2-30 micrometers preferably from this point.

[0039] electrolytic plating -- said film configuration -- the shape of Webb -- the shape of a non-edge pipe -- even when -- although essentially performed by the electrolytic solution which a difference does not have and contains a conductive metal by using as cathode the conductive metal thin film layer (2B) which is a substrate layer, it is the range where the plating condition is generally also performed. For example, when a metal thickness membrane layer (3) becomes with copper, as the electrolytic solution, it is immersed in the plating bath which added few \*\* of a brightener to this by using a copper sulfate and a sulfuric acid as a principal component, and the degree of bath temperature of about 20-40 degrees C, cathode-current-density about one to 7 A/dm<sup>2</sup>, the anode plate / cathode surface ratio 1:1, air stirring, and filtration (eddy condition) are always performed. Plating time amount is proper by prior conditioning. Moreover, when becoming with nickel, others are performed in the almost same condition range as the above using the plating bath which uses a nickel sulfate, a nickel chloride, and a boric acid as a principal component. In addition, before performing this electrolytic plating, it is good to carry out cleaning washing of the front face of a conductive metal thin film layer (2B).

[0040] By coming to have the property which was further excellent compared with the former, the M-PI film which becomes with the above is various, and comes to be used abundantly more. For example, more, with fine patternizing, the FPC substrate field of which higher thermal resistance is required is begun, and it is effectively used as fixing vessel part material (the shape of the shape of a roll, and a belt) which took in the electromagnetic-induction heating method of a color copying machine. In addition, in the use as fixing vessel part material of this color copying machine, surface coating processing by fluororesin is further performed on this endless tubing-like film for grant of a higher mold-release characteristic.

[0041]

[Example] Below, it explains in full detail further according to an example with the example of a comparison. In addition, the detachability and coefficient of linear expansion in this example are measured by the following approach.

- Lay [ firmly ] that it is also at tension 2 N/cm about a detachability profit \*\*\*\* endless tubing-like M-PI film across two rotation rollers (30mm diameter). By heating (the source of heating is built in one of this rotation roller, and the temperature is controlled by 150 degrees C), and rate 20rpm, it continues rotating at

150 degrees C for 5 hours, it stops at them, repeat exfoliation (rapid exfoliation) of the same location is carried out 3 times with a Scotch tape (trademark), and the existence is seen.

- Coefficient of linear expansion (cm/cm/degree C)

Each film equivalent to PI insulating layer which forms each class, and PI semi-conducting layer is produced, and it measures the following condition about each by making this into a sample. Thermal-analysis system TA-50WS [ measuring instrument ] of apparatus-for-thermomechanical-analysis TMA-50 by Shimadzu Corp. and a sample were die length of 10mm cut into the hoop direction, and the range of this die length, and the range of measurement temperature was 100-200 degrees C, and it was measured continuously, carrying out a temperature up the rate for 5-degree-C/. The coefficient of linear expansion in obtained each point was averaged and computed, and it considered as this multiplier.

[0042] (Example 1) The polycondensation reaction of 3, 3', 4, and this molar quantity of 4'-biphenyl tetracarboxylic dianhydride and p-phenylene diamine was first carried out at 20 degrees C among the NMP solvent, and 3kg of aromatic series PA acid solutions 1 (solution viscosity 1.8 Pa-s) of 13.5 % of the weight of solid content concentration was compounded. And this 1kg was extracted, preliminary mixing was carried out, first stirring boric-acid aluminum whisker (coefficient-of-linear-expansion  $4.2 \times 10^{-6}$  cm/cm/degree-C, specific gravity 3.6 [ about ]) 3.0g with an agitator with a wing as microfilament fine particles A to this, this was further moved and changed to the ball mill, and mixed distribution was fully carried out. This is called the shaping raw material 1 below. In addition, when these some PA acid solutions 1 were extracted, and removal and imide-izing of a solvent were performed, it considered [ stoving of this was cast and carried out to the glass plate, and ] as the polyimide film and this coefficient of linear expansion was measured, it was  $1.8 - 2.2 \times 10^{-5}$  cm/cm/degree C.

[0043] Centrifugal molding was performed the following condition using said obtained following shaping raw material 1, and it fabricated on the non-edge pipe-like PA acid film first, and the A-PI film of said whisker surface maldistribution was obtained.

[0044] Centrifugal molding conditions are as follows. It is the thing of structure explained in the text as shaping equipment, and conditions were carried out as follows.

O Metal drum .. Width of face of 450mm, the bore of 360mm, the amount of supply of O shaping raw material 1 .. Having started rotation of this drum slowly and moving a slit nozzle to the left from the right according to the rate, a total of this 275g undiluted solution 1 was supplied, and it applied to the perimeter side.

O Rotational speed and heating .. While starting heating toward 120 degrees C, rotational speed was gradually gathered toward the rate of 18 rad/s. After reaching this temperature and this rate, rotation heating was carried out for 120 minutes. This film with free-standing was fabricated by this drum inner skin.

[0045] And said obtained non-edge pipe-like PA acid film was exfoliated from the metal drum, this was fitted in hollow tubular metal mold with an outer diameter [ of 353mm ], and a width of face of 400mm, and this was put into hot air drying equipment, and 90 minutes was required, and it heated to 450 degrees C, and heated for 30 minutes at the temperature further. It cooled, and it took out from this dryer, and it de\*\* (ed) and the non-edge pipe-like A-PI film was obtained. The thickness of this obtained film is 50 micrometers, and an outer diameter is 353mm, and a part of edge was cut, when observing this cross section in SEM, the boric-acid aluminum whisker was observed by the surface part (field with a thickness of about 3 micrometers), and inside, it did not see at all.

[0046] And sputtering was carried out to the front face of said said obtained A-PI film (they are cut finishing and the outer diameter of 353mm to the size and width of face of 360mm) the following condition, and thin film layer 2B of nickel was formed in it.

O Sputtering system .. Although indicated in the outline text, on the turntable, the cylinder object (the outer diameter of 352mm, height of 370mm) of a piece was fixed, and this film was attached in this.

O Spatter conditions .. The target stood face to face against this film perpendicularly, and fixed the nickel plate with a width of face [ of 100mm ], and a die length of 380mm to the wall surface, and the spatter of the degree of vacuum was carried out for 15 minutes by output voltage 6.5 W/cm<sup>2</sup>, the distance of 100 degrees C, this target, and this film plane rotating 15mm and this turntable the rate for 1m/whenever [ 10-3Torr and vacuum chamber internal temperature ] in an argon permutation.

[0047] It was 500nm, when nickel thin film layer 2B was extremely formed in homogeneity and the thickness of this layer was measured in said A-PI film front face. And exfoliation was absolutely none although the repeat peel test of the same location was carried out 3 times with the Scotch tape.

[0048] Next, on nickel thin film layer 2B of said obtained film, electrolytic plating was performed the following condition and the laminating of the electric conduction thick-film layer 3 of nickel was carried

out. The Watts bath nickel liquid by which temperature control was carried out to 30 degrees C was used as the electrolytic solution, this film supported by this in the shape of a cylinder was immersed, and eddy electrolysis was performed for 10 minutes by cathode-current-density 5 A/dm<sup>2</sup>. When finishing, the whole was fully washed, and it dried and was made the product. Although this whole film changed into the condition hard a little, it was the last hardness which would become a problem for crookedness in any way. The nickel layer by which the laminating was carried out was uniform without nonuniformity, and thickness was 20micrometer\*\*0.5micrometer (20-place measurement average). And both the sides of this M-PI film were cut 15mm, and detachability was tested on said conditions about this. Although Scotch tape exfoliation was tried over 20 places as a result, there was no exfoliation. In addition, surface resistivity was a 10-3ohm/\*\* digit. In addition, this non-edge pipe-like M-PI film is illustrated by drawing 1. A branch number (1b) shows this whole film with a perspective view, (1c) is the D-D sectional view, and, as for the A-PI film of boric-acid aluminum whisker 1a surface maldistribution, and 2B, 1 is [ a nickel thin film layer and 3 ] nickel thick-film layers.

[0049] (Example 2) The polycondensation reaction of this molar quantity of pyromellitic acid 2 anhydride, and 4 and 4'-diamino diphenyl ether was carried out at 20 degrees C among the NMP solvent, and 3kg of aromatic series PA acid solutions 2 (solution viscosity 1.9 Pa-s) of 13.7 % of the weight of solid content concentration solid content concentration was compounded. And this 1kg was extracted, preliminary mixing was carried out, first stirring potassium titanate whisker (coefficient-of-linear-expansion 6.8x10<sup>-6</sup> cm/cm/degree-C, specific gravity 3.3 [ about ]) 5.0g with an agitator with a wing as microfilament fine particles A to this, this was further moved and changed to the ball mill, and mixed distribution was fully carried out. This is called the shaping raw material 2 below. In addition, when these some PA acid solutions 2 were extracted, and removal and imide-izing of a solvent were performed, it considered [ stoving of this was cast and carried out to the glass plate, and ] as the polyimide film and this coefficient of linear expansion was measured, it was 2.2 - 2.4x10<sup>-5</sup> cm/cm/degree C.

[0050] Centrifugal molding was performed the following condition using said obtained following shaping raw material 2, and it fabricated on the non-edge pipe-like PA acid film first, and the A-PI film of said whisker surface maldistribution was obtained.

[0051] Centrifugal molding conditions were performed on the same conditions as said example 1. PA acid film of the shape of endless tubing which has free-standing in the same condition as an example 1 was fabricated by metal drum inner skin.

[0052] And the aforementioned metal drum was removed from the rotation roller, and it put into hot air drying equipment as it is, and 90 minutes was required, and it heated to 400 degrees C, and heated for 30 minutes at that temperature further. It cooled and took out from this dryer, and it exfoliated and the non-edge pipe-like A-PI film was obtained. The thickness of this obtained film is 51 micrometers, and an outer diameter is 353mm, and a part of edge was cut, when observing this cross section in SEM, the potassium titanate whisker was observed by the surface part (field with a thickness of about 4 micrometers), and inside, it did not see at all.

[0053] Next, sputtering was carried out to the front face of said said obtained A-PI film (they are cut finishing and the outer diameter of 353mm to the size and width of face of 360mm) the following condition, and copper thin film layer 2B was formed in it. except for considering as copper as a target and considering as 6.2 W/cm<sup>2</sup> as output voltage -- an example 1 -- it was the same and the spatter was carried out.

[0054] It was 500nm, when copper thin film layer 2B was extremely formed in homogeneity and the thickness of this layer was measured in said A-PI film front face. And exfoliation was absolutely none although the repeat peel test of the same location was carried out 3 times with the Scotch tape.

[0055] Next, on copper thin film layer 2B of said obtained film, electrolytic plating was performed the following condition and the laminating of the copper thick-film layer 3 was carried out. This film supported in the shape of a cylinder was immersed in the electrolytic solution of the copper by which temperature control was carried out to 25 degrees C, and eddy electrolysis was performed for 7 minutes by cathode-current-density 1 A/dm<sup>2</sup>. When finishing, the whole was fully washed, and it dried and was made the product. Although this whole film changed into the condition hard a little, it was the last hardness which would become a problem for crookedness in any way. The copper layer by which the laminating was carried out was uniform without nonuniformity, and thickness was 8\*\*1.0 micrometers (20-place measurement average). And both the sides of this M-PI film were cut 15mm, and detachability was tested about this. Although exfoliation was tried over 20 places, there was no exfoliation. In addition, surface resistivity was a 10-4ohm/\*\* digit.

[0056] (Example 3) 1kg was extracted from the aromatic series PA acid solution 1 obtained in the example

1, first stirring 3.0g and 20.0g (volume-resistivity 10-1 ohm-cm) (it being 12.7 % of the weight to solid content) of CB fine particles of this example and the same boric-acid aluminum whisker with an agitator with a wing to this, it added and preliminary mixing of them was carried out at it, this was further moved and changed to the ball mill, and mixed distribution was fully carried out. This is called the shaping raw material 3 below.

[0057] And centrifugal molding was first carried out using said shaping raw material 3. Although the centrifugal molding conditions in this case followed the example 1, rotational speed of a metal drum was made into 13 rad/s, and rotation and heating time were set as for 140 minutes. This film with free-standing was fabricated by this drum inner skin.

[0058] And it exfoliated from the metal drum, and it fitted in hollow tubular metal mold on conditions as well as an example 1, and said obtained non-edge pipe-like PA acid film was put into hot air drying equipment, and was heated. It cooled, de\*\* (ed) from this metal mold, and the non-edge pipe-like A-PI film was obtained. The thickness of this obtained film is 57 micrometers, an outer diameter is 353mm, and it was observed that an assembly and CB fine particles will also be \*\*\*\* (ed) by the surface part also into this whisker part, and the boric-acid aluminum whisker will distribute [ the part ] to the whole interior like an example 1 at homogeneity if a part of edge is cut and this cross section is observed in SEM. And when [ of a front face and a rear face ] surface resistivity (applied-voltage 250V) measurement was carried out, they were  $1.3 \times 10^{12}$  ohms / \*\*, and  $1.1 \times 10^{12}$  ohm/\*\* respectively. It turns out that CB fine particles are distributed by homogeneity at the whole from now on.

[0059] And sputtering was carried out to the front face of said obtained A-PI film (the size and width of face of 360mm, outer diameter of 353mm) the following condition, and copper thin film layer 2B was formed in it. Except setting spatter time amount as for 20 minutes, spatter conditions were the same as the example 2, and were performed. It was 600nm, when copper thin film layer 2B was extremely formed in homogeneity and the thickness of this layer was measured. And exfoliation was absolutely none although the repeat peel test of the same location was carried out 3 times with the Scotch tape.

[0060] Next, on thin film layer 2B of the copper of said obtained film, electrolytic plating was performed the following condition and the laminating of the copper thickness membrane layer 3 was carried out. Except setting time amount as for 10 minutes, electrolytic plating conditions were the same as the example 2, and were performed. When the copper thickness membrane layer 3 was formed that there is no nonuniformity in homogeneity very much and the thickness of this layer was measured, it was  $10^{**} 1.3$  micrometers (20-place measurement average). And although detachability was tested like [ film / this / M-PI ] the example 2, and exfoliation was tried over 20 places, there was no exfoliation.

[0061] (Example 1 of a comparison) 1kg of the aromatic series PA acid solution 2 obtained in the example 2 was extracted, mixed distribution of the potassium titanate whisker was fully similarly carried out on conditions, centrifugal molding was carried out on the same conditions as this example by having made this into the shaping raw material, it heated similarly with hot air drying equipment succeedingly, full deliquoring and imide-ization were performed, and the A-PI film of this whisker surface maldistribution was obtained.

[0062] And electroless deposition of nickel was performed on all the front faces (said whisker side) of said A-PI film the following condition. First, after being immersed in the 90-degree C caustic soda water solution (20 g/l) for 5 minutes, rinsing desiccation of the front face of this film was fully carried out, and surface pretreatment was performed. Next, at the room temperature of the water solution which uses a palladium chloride, tin chloride, and a hydrochloric acid as a principal component, it was immersed for 10 minutes, this was pulled up, rinsing desiccation was fully carried out, and this was further immersed in the hydrochloric-acid water solution (removal of tin sake). This front face adheres to palladium and it has become a catalytic activity nucleus for no electrolyzing [ which is performed to a degree ].

[0063] And temperature control was carried out to the temperature of 35 degrees C by having made a nickel sulfate, hypophosphorous acid soda, and citric-acid Amon into the electroless deposition bath, and the aforementioned processing A-PI film was immersed in this for 10 minutes. Rinsing desiccation was fully carried out.

[0064] Next, it was immersed in Watts bath nickel liquid on the same conditions as an example 1 by having used said obtained nickel electroless deposition layer as cathode, and electrolytic plating of nickel was performed. The thickness of the nickel layer by which the laminating was carried out was  $24.7^{**} 3.7$  micrometers.

[0065] Since exfoliation would start partially [ a nickel layer ] in 30 minutes after rotation initiation and all would be in the desquamative state mostly in 60 minutes when the detachability test same about said



obtained tubular film as an example 1 was performed, this test was stopped. When the condition of isolation was seen, it was exfoliation between a substrate (A-PI film) and a nickel non-electrolyzer.

[0066] (Example 4) (example of an electromagnetic-induction heating test)

Using the shaping raw material 3 first prepared in the example 3, according to this example, electrolytic plating of the sputtering -> copper of centrifugal molding -> hot-air-drying -> copper was performed, and the same copper metalization M-PI endless tubular film as this example was obtained.

[0067] On the other hand, using the copolymer (melting point of about 305 degrees C) of tetrafluoroethylene and perfluoroalkyl vinyl ether, carrying out inner sizing (temperature of 80 degrees C) from an annular dice (temperature of 380 degrees C), melting extrusion was carried out and the non-edge pipe-like fluororesin film with 60\*\*7 micrometers [ in thickness ] (extension is 1.1 times to a lengthwise direction) and a bore of 355mm was fabricated.

[0068] And said copper metalization M-PI endless tubular film was attached in the hollow tubular metal mold used in the example 1, and said non-edge pipe-like fluororesin film was put on this, and it put into vacuum hot air drying equipment, and heated for 30 minutes at 120 degrees C. Contraction adhesion was able to be carried out firmly, eliminating oxygen, without this fluororesin film oxidizing a copper layer with this vacuum heating. And the temperature up was carried out to 360 more degrees C, and it heated for 10 minutes. It cooled in ordinary temperature and took out from this dryer. Melting adhesion of this fluororesin film was firmly carried out to the copper layer side of this M-PI endless tubular film. It is called a fluororesin covering PI film below.

[0069] And the belt rotation condition was equipped with said fluororesin covering PI film, the electromagnetic-induction exoergic coil has been arranged in this belt, the power of 120W was impressed, and whenever [ this hair side of belt surface temperature ] was measured. As a result, it generated heat immediately, it amounted to 160 degrees C, and became fixed. It has also checked generating heat effectively according to an electromagnetic-induction operation. In addition, since a mold-release characteristic with paper is also required, being what put the fluororesin here and having performed this test performs toner fixing by assuming the condition of having given the function. Although it is in the covering approach of this resin variously, this approach using the thing which was performed here and which was beforehand used as the tube is more effective.

[0070]

[Effect of the Invention] Since this invention is that a physical thin film form method and electrolysis plating, such as sputtering, combine especially on PI system film with which it comes to carry out maldistribution distribution of the aforementioned matter of passage specification on a front face and is this film by which surface electric conduction metalization was carried out, it does the following effectiveness so.

[0071] The adhesion of a surface electric conduction metalization layer and said PI system film plane was able to be raised sharply. This adhesion is natural when this film is a flat-like, but even if it has the adhesion force which had the shape especially of a non-edge pipe, was in the belt rotation condition, and has been improved by practical use level even if used under heating, it is.

[0072] Although mixed distribution of said special material is carried out at said metalization PI system film, it can \*\*taking a whole configuration, without it seeming that own essential character of PI film is reduced because the distributed condition is a surface part.

[0073] Since it is formed as even when it is quality, the electric conduction thin film layer by sputtering etc. can also form more thinly high performance and the conductive layer by the part electrolytic plating. When using it as for example, a FPC substrate, this can make line breadth of a circuit thinner and leads to more circuit accumulation.

---

[Translation done.]



\* NOTICES \*

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

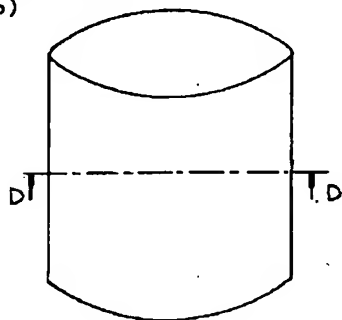
---

DRAWINGS

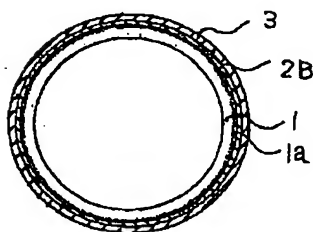
---

[Drawing 1]

(1 b)



(1 c)



D-D断面

---

[Translation done.]

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**